

Serial No. 09/831,915

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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
THOMAS DANIEL, ET AL. : EXAMINER: METZMAIER, D. S.
SERIAL NO: 09/831,915 :
FILED: MAY 25, 2001 : GROUP ART UNIT: 1712
FOR: HYDROGELS CAPABLE OF :
ABSORBING AQUEOUS FLUIDS :

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR,

Now comes Dr. Manfred Essig who deposes and states:

1. That I am a graduate of the University of Kaiserslautern and received a Ph.D. degree in the year 1981.
2. That I have been employed by BASF SE (formerly BASF Aktiengesellschaft) for 23 years as a scientist in the field of Electron Microscopy.
3. That the following experiments were carried out by me or under my direct supervision and control.

OVERVIEW

The experiments relate to a hydrogel according to the present invention.

A sample of Hysorb P7710 which is a dried hydrogel according to the present invention (see the Declaration by Dr. Thomas Daniel) was investigated by SEM (Scanning Electron Microscopy) and EDX (Energy-dispersive X-ray spectroscopy) to show that the silicon corresponding to the alkali metal silicate is distributed throughout the dried hydrogel particles and not just on the surface of the particles.

SAMPLE PREPARATION

In preparation of the measurement, gelatine capsules were filled with the Hysorb P7710 polymer particles. The gelatine capsules were cylindrical with a diameter of about 8 mm. In addition, after the Hysorb P7710 was already in the capsules, a polymer (commercially available LR-White, Agar Scientific Limited, Essex England) was filled into the capsules. After hardening of the LR-White polymer in the capsules, two of the gelatine capsules were included in a resin cylinder with about 25 mm diameter. The cylinder was then cut with a diamond rotary cutter at about 20 000 rpm. The reason that two capsules were used was to increase the probability of cutting polymer particles *more or less through the center* when cutting the resin cylinder. The cutting exposed a cross sections of the dried hydrogel particles. In order to make the surface electrically conductive, a thin film of carbon was deposited by vapor deposition on the cross section surface.

EXPERIMENTS

The experiments were performed using SEM and EDX. The SEM allowed generation of images of the cross-section surface of the sample and illustrated primarily the structure of the surface when employing secondary electrons. The exploitation of backscatter electrons gave

information about the structure and in addition information about the density of the material at the sample surface.

The method of differentiating between different elements was EDX (Energy-dispersive X-ray spectroscopy). In the EDX method, atoms are excited with X-ray radiation. The excited atoms then emit X-rays which are specific for the respective element and which are measured.

A standard SEM method was used for the experiments. The cross-section sample as prepared was put on the stage of a SEM microscope (company LEO, model DSC 960). The standard imaging parameters were 5kV for the secondary electron images and 20 kV for the back scatter electron images.

For analytical purposes an EDX spectrometer was adapted to the scanning microscope. The system was from the company Röntec in Berlin, Germany.

A 20kV primary beam energy was used for the generation of the elemental distribution images obtained by EDX. In order to enhance the contrast when depicting the images with EDX in the report, only signal strengths are presented which were representative for local concentrations above 50% of the average concentration in the sample. This focus on certain signal strengths improved correlation of the various elemental pictures and made the images easier to interpret.

RESULTS

The analysis of the images was based on the most clearly visible particles of SAP. The field of view was representative for the whole cross section of the sample which was checked visually.

A sheet of three pictures, attached herewith, is incorporated into the Declaration by reference.

The first picture from the top is a backscatter electron image and shows the location of higher densities brighter than the vicinity having a lower density of the material at the sample cross section. The darkest areas (black) represent the polymer (commercially available LR-White, Agar Scientific Limited, Essex, England) used during preparation of the samples.

Therefore, due to the known composition of the sample, the bright areas can be interpreted as silicon rich areas. This becomes evident when comparing with the element map of the silicon (the last picture at the bottom in red color) with the first picture. The bright areas in the first picture correlate to the red areas in the last picture, with a higher brightness representing a higher concentration of silicon. Another information is that the silicon is found within the cross-section of the particles.

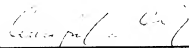
The second picture from the top shows the elemental distribution of sodium in the cross section of the particles, as indicated by the yellow color. Since sodium is contained in the silicate as well as the Hysorb P7710, the second picture cannot be used to further identify the location of the silicate. In fact, the concentration of the sodium from Hysorb P7710 is higher than from the silicate itself.

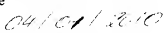
As mentioned, the third picture shows the elemental distribution of silicon in the cross section of the particles. Clearly, the third picture shows that silicon is present throughout the particle and not just on the surface.

The dried hydrogel particles of the present invention are different from those of WO 97/46189 because the silicon is found throughout the entire particle and not just on the surface.

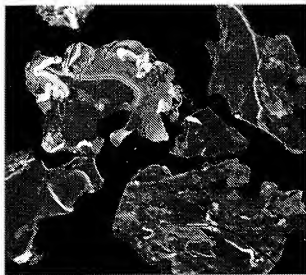
4. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing therefrom.

5. Further deponent saith not.



Signature


Date

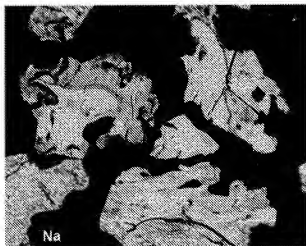


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200μm

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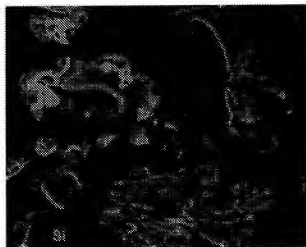
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Na

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Abb: 01 Na



Si

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